Uniform Micro-Particles as Reference Material for Mass Spectrometry

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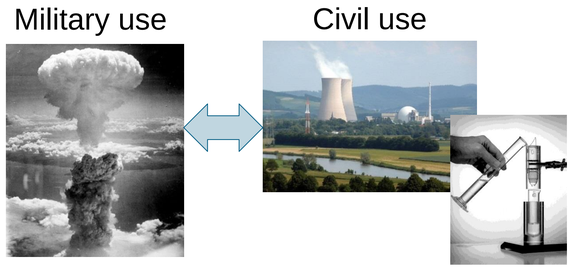
Nuclear Safeguards

Dual-use nature of nuclear technology requires attention to non-proliferation.

Military use ⟷ Civil use



Non-Proliferation Treaty (NPT):
- In force since 1970, 190 members;
- IAEA controls the peaceful use through “Safeguards”.

Nuclear safeguards:
- Verification measures to deter states from the misuse of nuclear material and facilities;
- Inspections of nuclear facilities by the IAEA.

Particle Analysis in Nuclear Verification

Swipe sampling supports the IAEA detection of undeclared activities.

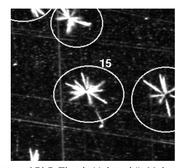
Source: IAEA

Detection of traces from enrichment and reprocessing activities through highly sensitive analysis of actinides.

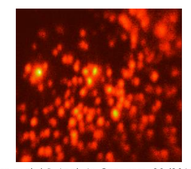
Analysis of isotopic ratios of actinides reveal material handling history of facility.

Due to possible consequences, very high precision measurements required.

Typical isotope analysis by:
- (Large Geometry) Secondary Ion Mass Spectrometry (LG-SIMS);
- (Fission Track) Thermal Ionization Mass Spectrometry (FT-TIMS).



B. Stetzer / PhD Thesis Universität Mainz (2001)



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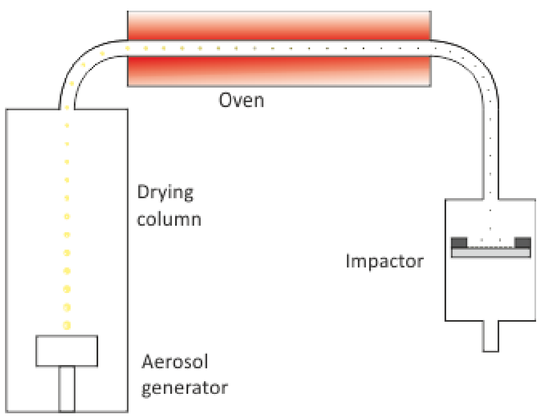
High need for relevant reference materials for quality control and quality assurance, but none available.

! Dedicated reference material for Quality Assurance and Quality Control required

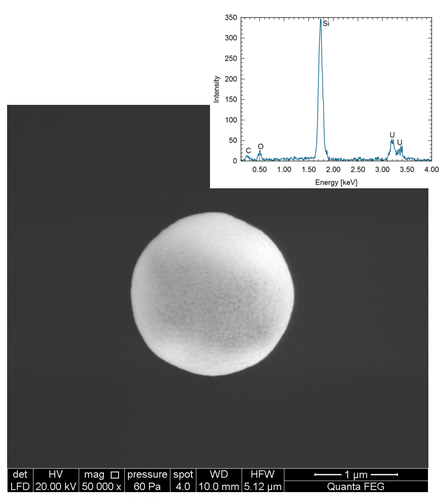
Particle Generation and Characterization



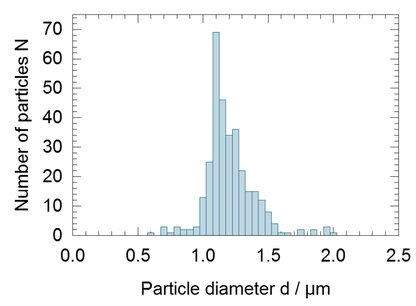
1) Uranyl nitrate/ethanol mixture with known isotopic composition and uranium content;
2) Generation of aerosol droplets with vibrating orifice aerosol generator (VOAG);
3) Drying and calcination of uranyl nitrate;
4) Collection of microparticles.

$$\text{Volume } [\text{m}^3] = \frac{\text{Feed rate } [\text{m}^3\ \text{s}^{-1}]}{\text{Frequency } [\text{s}^{-1}]}$$

$$\text{Elemental mass } [\text{kg}] = \text{Volume } [\text{m}^3] \cdot \text{Concentration } [\text{kg m}^{-3}]$$



SEM micrograph



Particle size distribution

Certification

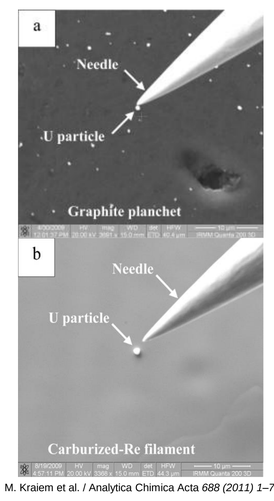
Certification of isotopic composition of particles, uranium content per particle as informative value (at first).

Investigation of stability and homogeneity of produced particles.

Isotopic composition:
- TIMS;
- (LG-)SIMS;
- (MC-)ICP-MS.

Uranium content:
- (ID-)TIMS;
- (ID-)ICP-MS.

Other informative values:
- Size/morphology (e.g. SEM);
- Crystal structure (e.g. µ-Raman);
- Chemical composition (e.g. EDX).



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Further Possibilities

Direct certification of collected particles on substrate
(e.g. on Si wafer or quartz disc)

Advantages:
+ No further treatment required.

Disadvantages:
- Inhomogeneous distribution;
- Single sample production.

Particles transferred from substrate into suspension
(e.g. in ethanol or water)

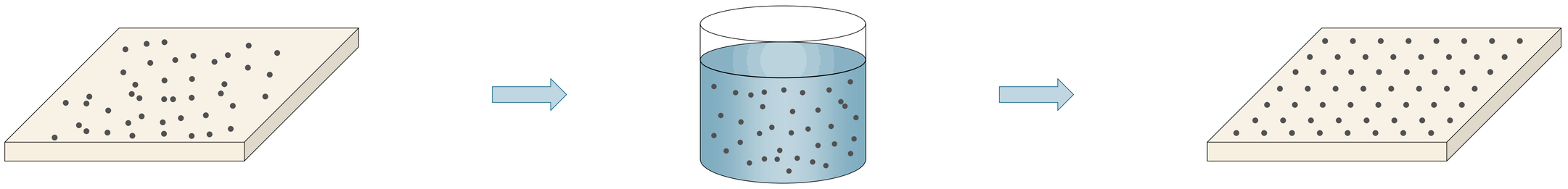
Advantages:
+ Flexibility;
+ Simplified sub-sampling.

Disadvantages:
- Particle dissolution possible;
- Aggregation and/or settling possible.

Particles embedded on synthesized substrate
(e.g. polyimide foil or epoxy resin)

Advantages:
+ Improved homogeneity;
+ Simplified large batch production.

Disadvantages:
- Specific substrate material;
- Possible dissolution during synthesis.



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